



PATENT

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

Applicants: Siepel et al.

Examiner: Tran, Lien

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For: INGREDIENTS FOR  
EXPANDED FOODS

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**DECLARATION UNDER 37 C.F.R. § 1.132**

The undersigned, Pieter L. Buwalda of Mondriaanstraat 32, Groningen, the Netherlands, herewith declares as follows:

1. I am a Food Starch Specialist at the Food Competence Center of the international co-operative AVEBE in Foxhol, The Netherlands, the world's largest manufacturers of potato starch derivatives. I took up this position on December 1 of 2001. Before that I was associated with the Chemistry Department of AVEBE for a period of almost twelve years where I performed research on various starch applications, the last five years mainly food oriented. My specialisation is Chemistry of Starch.

I hold a PhD degree in Organic Chemistry from the University of Groningen, the Netherlands, and have written a number of publications and am a co-inventor of various patents relating to Starch Chemistry. In 1997, for instance, I acted as an author on Granular and Molecular Structure of Starch, The 3<sup>rd</sup> CAFST International Symposium, page 109. A list of publications is attached to this declaration.

2. I am co-inventor of the patent application as identified above. The invention of this application is based on the insight that a foodstuff that is prepared with the use of an amylopectin root/tuber starch in a process involving heating to a temperature above the glass transition temperature of the starch exhibits unexpectedly high expansion as compared to a similar foodstuff prepared from regular (i.e. amylose containing) potato starch or amylopectin maize starch (waxy maize). A foodstuff prepared with such a starch moreover has an advantageous texture.

3. Jeffcoat et al. show that amylopectin potato starch derivatives are much higher in viscosity than waxy maize derivatives (see col. 2, lines 30-35 and 43-48, as well as Fig. 1 and Tables II and III). It is generally understood to those skilled in the art that expansion is inversely related to viscosity (the higher the viscosity, the lower the expansion).

Kokini et al. derived the following expression for bubble growth rate (i.e., expansion):

$$dR/dt = R\ddot{A}P/\eta$$

where  $R$ ,  $\eta$ , and  $\ddot{A}P$  are respectively the bubble radius, melt viscosity, and the vapour pressure difference between the interior of a bubble and the surroundings (J.L. Kokini, C.N. Chang, L.S. Lai, "The role of rheological properties on extrudate expansion", Food Extrusion Science and Technology (J.L. Kokini, C.T. Ho, M.V. Karwe, Eds.), Marcel Dekker, New York (1992), p. 631-652).

4. Therefore, it would be expected that use of an amylopectin potato starch as disclosed by Jeffcoat et al. in the procedure of van Hulle et al. would lead to reduced expansion when compared to waxy maize starch or regular potato starch.

5. The degree of expansion found in accordance with the invention is surprising. Expansion experiments have been carried out comparing amylopectin potato starch, crosslinked amylopectin potato starch and waxy maize starch. The experimental procedures used are described in paragraph 6.

## 6. Experimental Procedures

### **Materials**

#### **Equipment:**

- Bühler-macaronipress, type FLPA
- Stephan high speed mixer, type UMB 25
- Sartorius moisture analyzer
- Bühler pellet grinder
- Fryland Deepfat fryer

#### **Ingredients:**

##### Starches Standard mix:

- |                                    |     |     |
|------------------------------------|-----|-----|
| - Potato granules Rixona, type SFG | 40% |     |
| - Starch                           |     | 52% |
| - Instant starch                   | 6%  |     |
| - Salt                             |     | 2%  |

#### Miscellaneous:

- Frying oil Remia fluid

#### **Starches to evaluate:**

- Native amylopectin potato starch (Eliane, Avebe)
- Waxy maize starch
- Crosslinked amylopectin potato starch (Eliane, Avebe), obtained by reacting 400 mg crosslinking agent (sodium trimetaphosphate) with starch.

#### **Methods:**

After having prepared the ready mixes in the Stephan-mixer, on the intended moisture content of about 36%, (controlled by Sartorius moisture analyzer) the obtained mixes have been extruded under standard conditions via the Bühler macaronipress. The drying of the cutted, screwtype pellets, was done under the following conditions: 4 hours at 70°C, at 20% R.H.. At least one day after drying, a part of the pellets has been grinded in the Bühler pellet grinder, in order to estimate the end moisture content of the pellets. The pellets have afterwards been fried in vegetable oil of Remia, during 10 seconds at 190°C and assessed.

#### **Analyses**

At least one day after production, the obtained end-products have been organoleptically assessed on standard properties (see Table). The density of the end products was measured in a calibrated beaker of 2L wide model and indicated in g/L. The expansion index is obtained by calculating the expansion in ml per gram.

## Results

	Native amylopectin potato starch	Waxy maize starch	Crosslinked amylopectin potato starch
Visual expansion (small/big 0-5)	2	1.5	2.5
Performance (smooth/not smooth 5-0)	2.5	1.5	2.5
Color (white/yellow/grey/brown)	y	yw	yb
Bite (hard/soft 0-5)	2	2	2.5
Crispiness (not/very 0-5)	2	1	3.5
Pore size (small/big 0-5)	1.5	1	2.5
Taste (potato-like/not 5-0)	5	2	5
Shape snacks (irregular/regular 0-5)	2	1.5	2.5
Density (g/L)	57	83	54
Expansion index (mL/g)	17.5	12.0	18.5

7. Thus, the expansion results of the experiment described in paragraph 6 are the following:

amylopectin potato starch: 17.5 mL/g

waxy maize starch: 12.0 mL/g

crosslinked amylopectin potato starch: 18.5 mL/g.

These results show the unpredictability of the expansion behavior of these starches. These results could not have been predicted based on the teachings of Jeffcoat et al. and Van Hulle et al.

8. The Examples of the present application, which were carried out in 1998 under my supervision, reflect the superior expansion characteristics of a foodstuff prepared in a process of the invention in comparison with a foodstuff prepared using regular potato starch or waxy maize starch (see Tables 1 and 2). In particular, Table 1 shows that the product prepared in Example 1, using native regular potato and pregelatinized waxy maize starch, shows an expansion rated a 6, whereas the product prepared in Example 4, using amylopectin potato starch and a pregelatinized amylopectin potato starch, shows an expansion rated an 8.

9. The results of more detailed expansion measurements are shown in Table 2. These measurements were performed by weighing the amount necessary to fill a 2 liter measuring cylinder with baked snacks prepared as described in Examples 5-11. The results

are expressed as the volume occupied by 200 grams of snacks. As can be seen in Table 2, 200 grams of the snacks prepared in Examples 5 and 8-9<sup>1</sup>, prepared using amylopectin potato starch, all occupy 2100 milliliters or more; whereas 200 grams of the snacks prepared in Examples 6 and 7, prepared using waxy maize starch and regular, amylose containing potato starch, respectively, occupy only 1880 and 1610 milliliters, respectively. In the worst case (i.e. comparing the results for waxy maize of Example 6 with those for amylopectin potato starch in Example 9), this still is an increase in expansion of more than 15%.

10. The method disclosed by van Hulle et al. does not result in a foodstuff with the beneficial properties and characteristics of the foodstuffs prepared by a method of the present invention. In particular, the desired expansion properties will not be obtained. Dough containing pregelatinized starch cooked in an extruder under pressure would not expand.

11. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true. Further that these statements were made with the knowledge that willfully false statements and the like so made are punishable by fine or imprisonment or both under Section 1001 of Title 18 of the United States Code, and that such willfully false statements may jeopardize the validity of the application of any patent issued thereon.

Respectfully submitted,

Dated: \_\_\_\_\_

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Pieter L. Buwalda

<sup>1</sup> Examples 10 and 11 should not be taken into account in this comparison because different recipes were used for preparing the snacks in these Examples. In particular, in Example 10 the starch dosage was increased by 50% and in Example 11 the water dosage was increased by 40%.

## List of Publications

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5. Buwalda, Pieter Lykle; Kesselmanns, Ronald Pieter Wilhelmus; Maas, Augustinus Arnoldus Maria; Simonides, Hylke Hotze. **Hydrophobic starch derivatives, their manufacture and uses.** PCT Int. Appl. (2000), 31 pp. CODEN: PIXXD2 WO 2000042076 A1 20000720 CAN 133:121916 AN 2000:493578 CAPLUS
6. Thirkow, Roelfina Willemina Antonia; Buwalda, Pieter Lykle. **Heat-stable high-amylopectin starch for use in baking.** PCT Int. Appl. (2000), 23 pp. CODEN: PIXXD2 WO 2000005973 A1 20000210 CAN 132:136689 AN 2000:98230
7. Buwalda, Pieter Lykle; Guns, Jacobus; Lacroix, Jacques. **Depilatory paint thickener based on starch for hides.** PCT Int. Appl. (2000), 60 pp. CODEN: PIXXD2 WO 2000005420 A1 20000203 CAN 132:124472 AN 2000:85057 CAPLUS
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9. van der Huizen, Adri A.; Buwalda, Pieter L.; Wilting, Theo; Pol, Harm; Jekel, Andries P.; Meetsma, Auke; van de Grampel, Johan C. **Preparation of urethane and urea derivatives of (NPCl<sub>2</sub>)<sub>3</sub>. Crystal structure of a spirocyclic phosphazene with a phosphacyanuric loop.** Journal of the Chemical Society, Dalton Transactions: Inorganic Chemistry (1972-1999) (1994), (4), 577-81. CODEN: JCDTBI ISSN:0300-9246. CAN 121:9686 AN 1994:409686
10. Van de Grampel, J. C.; Alberda van Ekenstein, G. O.-R.; Baas, J.; Buwalda, P. L.; Jekel, A. P.; Oosting, G. E. **Preparation and polymerization of styrene-, acrylate-, and methacrylate-substituted cyclophosphazenes.** Phosphorus, Sulfur and Silicon and the Related Elements (1992), 64(1-4), 91-8. CODEN: PSSLEC ISSN:1042-6507. CAN 116:256116 AN 1992:256116 CAPLUS
11. Buwalda, Pieter L.; Steenbergen, Andre; Oosting, Gerard E.; Van de Grampel, Johan C. **The addition of phosphazeneocuprates to aldehydes and ketones: a new route to gem-organo-substituted cyclotriphosphazenes.** Inorganic Chemistry (1990), 29(14), 2658-63. CODEN: INOCAJ ISSN:0020-1669. CAN 113:78690 AN 1990:478690 CAPLUS

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